FOS/TAC

Introduction of new Digester Metrics

UOSA

54 MGD
Advanced Water Reclamation Facility - Indirect Potable Reuse

- Conventional
- Chemical
- Physical/Carbon
- Disinfection
- Carbon Regeneration
- Digestion
- Pelletizer
- Landfill
- Diesel Generators
- Cogen Facility

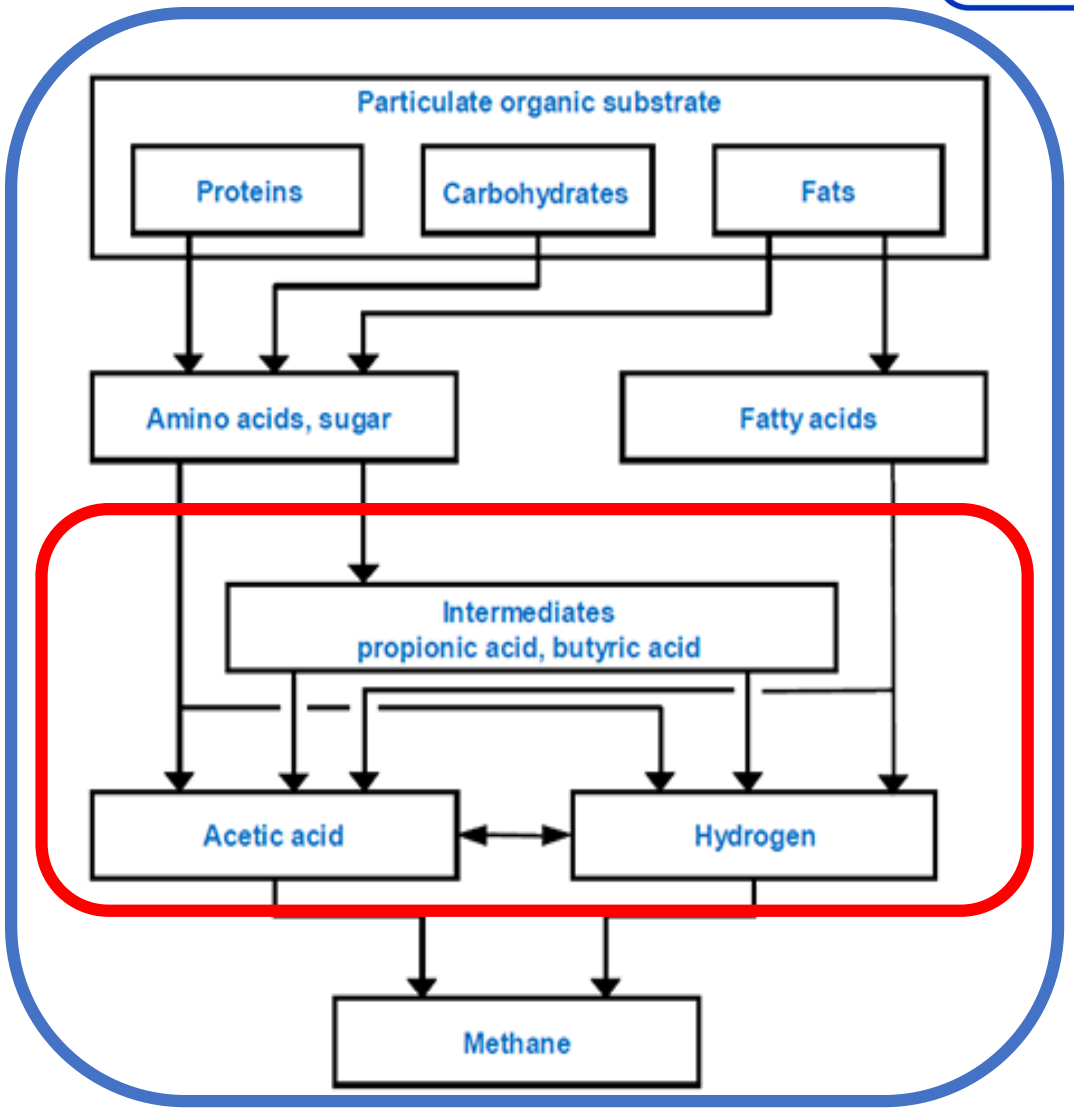
OCCOQUAN UPPER SERVICE AUTHORITY

Cogeneration Facility

FOS/TAC - Anaerobic Process Analytics

Potentiometric titration is an affordable and accurate method. In this case it is used to determine:

- **Content of Volatile Acids**
- **Digester Buffer Capacity**



FOS/TAC Ratio

The FOS/TAC ratio is an indicator for assessing fermentation processes.

The TAC value is an estimation of the buffer capacity of the sample and the FOS value corresponds to the volatile fatty acids content. It is calculated empirically according to the Nordmann method. (Hach DOC316.52.93087)

Titration - Method

A sample of 5 mL of fermentation substrate is titrated by 0.1 N of sulfuric acid solution (H_2SO_4) up to pH 5.0 to calculate the TAC value, expressed in mg/L of calcium carbonate ($CaCO_3$). Then the FOS value is obtained after a second titration step between pH 5.0 and pH 4.4. It is expressed in mg/L of acetic acid (CH_3COOH).

The Nordmann method for FOS determination is based on experiments with 20 mL of sample and 0.1 N H_2SO_4 of solution. The formula is adapted with a 5 mL sample. It is important to perform the titrations as close as possible to these conditions. (Hach DOC316.52.93087)

FOS/TAC - History of Nordmann method

1968 - McGhee, Method for Approximation of the Volatile Acid Concentrations in Anaerobic Digesters

Test series to determine fatty acid concentration and buffer capacity using acid consumption per changes of pH value.

1977 – Nordmann published paper that described method for monitoring of anaerobic digestion processes in wastewater applications.

Developed titration curves for waste water treatment plants within the limits described by McGhee.

Other AD titration tests have been developed as well. Some have different and more titration endpoints. Goal to increase exactness of the method. Authors: Jenkins, 1983 / Kapp, 1984 / Anderson and Yang, 1992 / Moosbrugger, 1993

Nordmann method found wide spread application in European AD-Industry. It represents an affordable method with quick return time for results.

FOS/TAC – VOA/Buffer capacity

TAC: H_2SO_4 consumption from start value to pH 5.0 is used to calculate buffer capacity of carbonate system. It is reported as mg/L calcium carbonate.

FOS: H_2SO_4 consumption from pH 5.0 to pH 4.4 expressed as sum of the below shown organic acids in mg/L acetic acid.

The pK_a values of the org. acids are between 5.0 and 4.4 The H_2SO_4 - acid dissociation constant is -3.9, therefore org. acids will be consumed by H_2SO_4 during titration and the H_2SO_4 volume can be expressed as sum of VOA.

Name	Ion	pK_a
Carbonic Acid	HCO_3^-	6.35
Acetic Acid	CH_3COO^-	4.76
Propionic Acid	$C_2H_5COO^-$	4.86
Valeric Acid	$C_4H_9COO^-$	4.84
Butyric Acid	$C_3H_7COO^-$	4.82

The method does not offer conclusions about the individual amount of each acid in the sample. The only acid that can be directly converted to CH_4 by anaerobic digestion is acetic acid. If the concentration of the other acids is high due to an inhibition other process parameter have to be consulted.

Sample Collection

Uniform sample collection practice should be insured:

- Sampling collection should not occur immediately after feeding, when batch feeding is used.
- With continuous feed schedule, sampling should be done daily at the same time.
- Well mixed digester content should be sampled. UOSA - 450 GPM side stream recirculation for sludge heating, samples are pulled from suction side of pumps.
- Change in sample location might lead to change in FOS/TAC.
- No airspace in sample bottles, immediate refrigeration or immediate sample processing at laboratory.
- When the lab detects a sample that is not conform with instructions, resampling of that sample is requested, with immediate refrigeration or processing of the sample.

Sample Processing

Samples should be checked for compliance with instructions before processing.

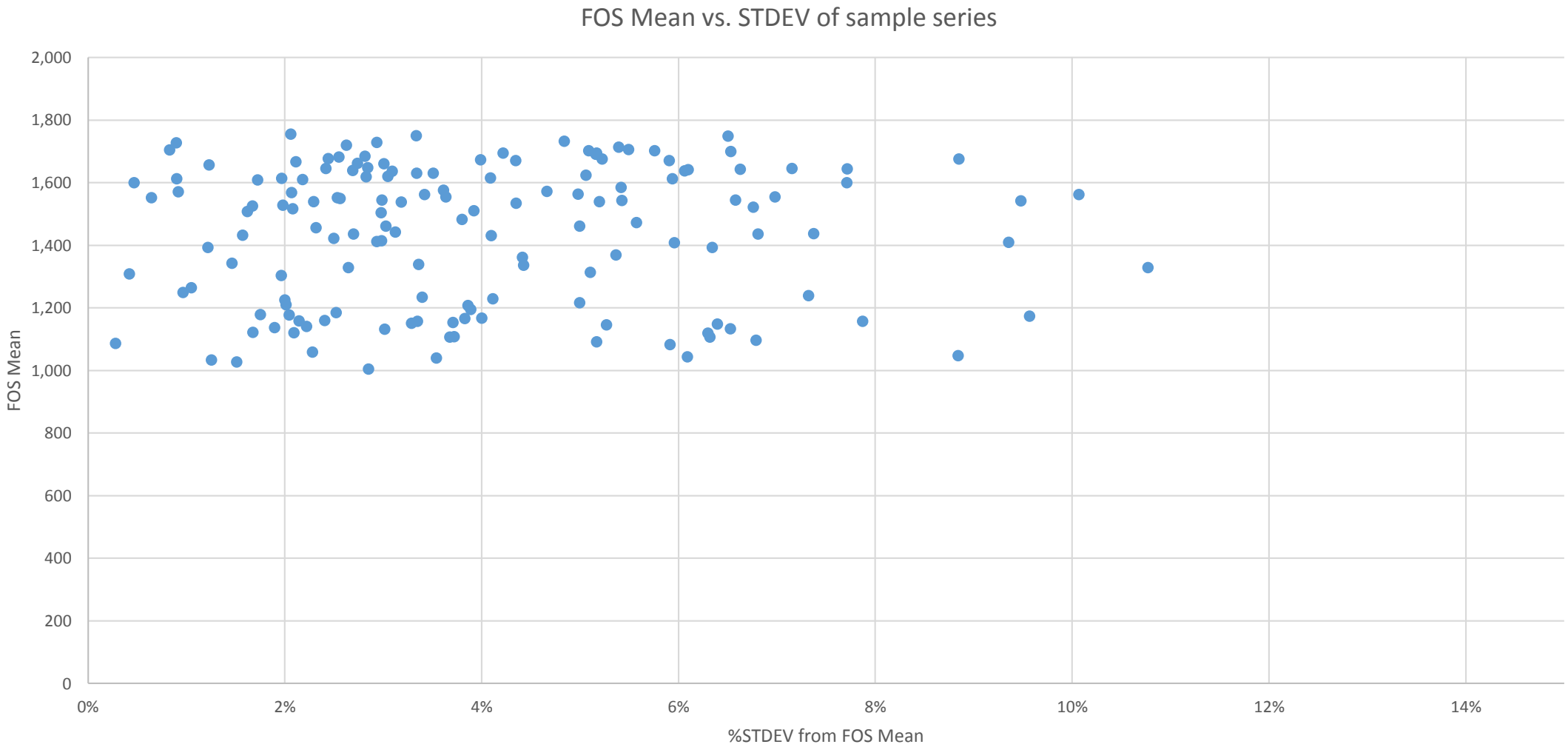
- Sample does not need to be stirred or shaken, just gently turned over.
- Nordmann method does not ask for sample filtration. Filtered samples lead different results. Filtration step would increase processing time and materials.
- In according to the applied method the sample volume i.e. 5 ml needs to be weighed out as 5g ± 0.1 g. Important during periods of foam in digester.
- Process one sample at the time. Do not prepare samples in order to save time. We saw increased deviations of results with increased sample holding times.
- Currently UOSA-lab is testing each sample three times and reporting the average value.

Data Reporting

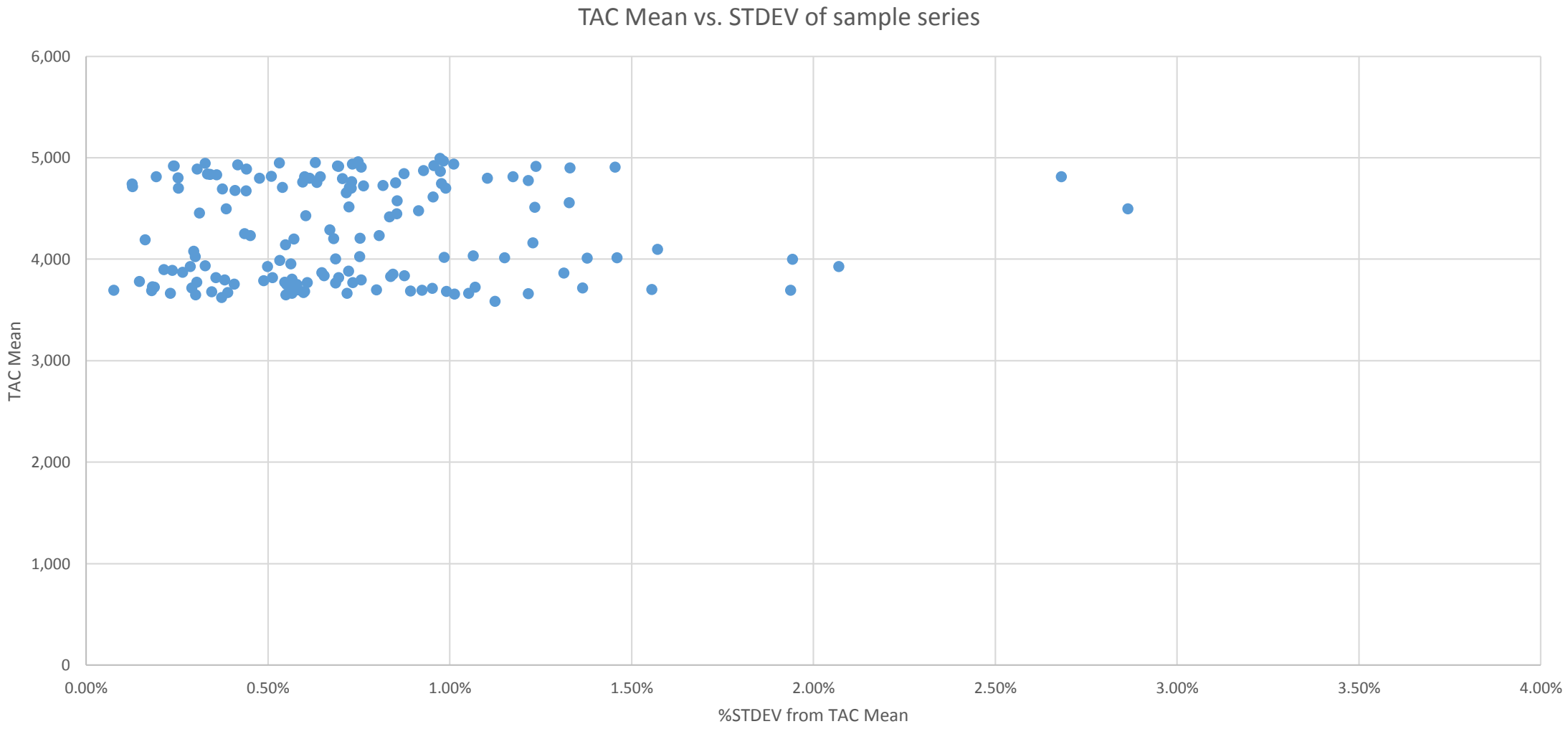
FOS-TAC Lab Data

	Prim Dig 1																		
Sample Date	Wt 1 (gm)	Wt 2 (gm)	Wt 3 (gm)	TAC 1	TAC 2	TAC 3	STEDV TAC 1-3	%STDEV from Mean	**TAC Mean**	FOS 1	FOS 2	FOS 3	STEDV FOS 1-3	STDEV	**FOS Mean**	FOS/TAC 1	FOS/TAC 2	FOS/TAC 3	**FOS/TAC mean**
7/5/16	5.01	5.03	5.00	4169	4045	4077	64	1.6%	**4097**	1286	1548	1392	132	9.4%	**1409**	0.308	0.383	0.341	**0.344**
7/6/16	4.96	5.02	4.97	3990	4078	3967	59	1.5%	**4012**	1391	1464	1450	39	2.7%	**1435**	0.349	0.359	0.365	**0.358**
7/7/16	5.04	5.04	5.04	4015	4020	4038	12	0.3%	**4024**	1288	1354	1342	35	2.6%	**1328**	0.321	0.337	0.332	**0.330**
7/8/16	4.91	4.95	4.99	3931	3947	3975	22	0.6%	**3951**	1456	1421	1385	36	2.5%	**1421**	0.370	0.360	0.348	**0.359**
7/9/16	4.92	4.91	4.92	3891	3895	3907	8	0.2%	**3898**	1307	1302	1313	6	0.4%	**1307**	0.336	0.339	0.336	**0.337**
7/10/16	4.92	4.92	4.96	3839	3867	3889	25	0.6%	**3865**	1305	1275	1326	26	2.0%	**1302**	0.340	0.330	0.341	**0.337**
7/11/16	4.99	5.00	5.03	3800	3844	3866	34	0.9%	**3837**	1564	1409	1440	82	5.6%	**1471**	0.411	0.367	0.373	**0.384**
7/12/16	5.02	5.00	5.00	3824	3781	3805	22	0.6%	**3803**	1321	1345	1360	20	1.5%	**1342**	0.345	0.356	0.358	**0.353**
7/13/16	5.02	4.99	5.00	4009	3979	3968	21	0.5%	**3985**	1720	1528	1673	100	6.1%	**1640**	0.429	0.384	0.422	**0.412**
7/14/16	4.99	5.02	5.01	3739	3771	3794	28	0.7%	**3768**	1457	1467	1640	103	6.8%	**1521**	0.390	0.389	0.432	**0.404**
7/15/16	4.99	5.00	4.99	3659	3703	3677	22	0.6%	**3680**	1375	1409	1392	17	1.2%	**1392**	0.376	0.381	0.379	**0.379**
7/16/16	4.95	4.90	4.98	3740	3676	3749	40	1.1%	**3722**	1287	1353	1373	45	3.4%	**1338**	0.344	0.368	0.366	**0.359**
7/17/16	5.00	4.94	4.95	3653	3661	3670	9	0.2%	**3661**	1274	1339	1392	59	4.4%	**1335**	0.349	0.366	0.379	**0.365**
7/18/16	4.99	5.01	5.01	3674	3704	3773	51	1.4%	**3717**	1418	1403	1284	73	5.4%	**1368**	0.386	0.379	0.340	**0.368**
7/19/16	4.99	5.02	5.01	3658	3667	3686	14	0.4%	**3670**	1549	1441	1537	59	3.9%	**1509**	0.423	0.393	0.417	**0.411**
7/20/16	4.99	5.00	4.97	3686	3686	3664	13	0.3%	**3679**	1387	1533	1460	73	5.0%	**1460**	0.376	0.416	0.398	**0.397**
7/21/16	4.99	4.95	4.99	3708	3645	3638	39	1.1%	**3664**	1318	1369	1490	88	6.3%	**1392**	0.355	0.376	0.409	**0.380**
7/22/16	5.01	5.00	5.00	3707	3701	3641	36	1.0%	**3683**	1524	1479	1518	24	1.6%	**1507**	0.411	0.400	0.417	**0.409**

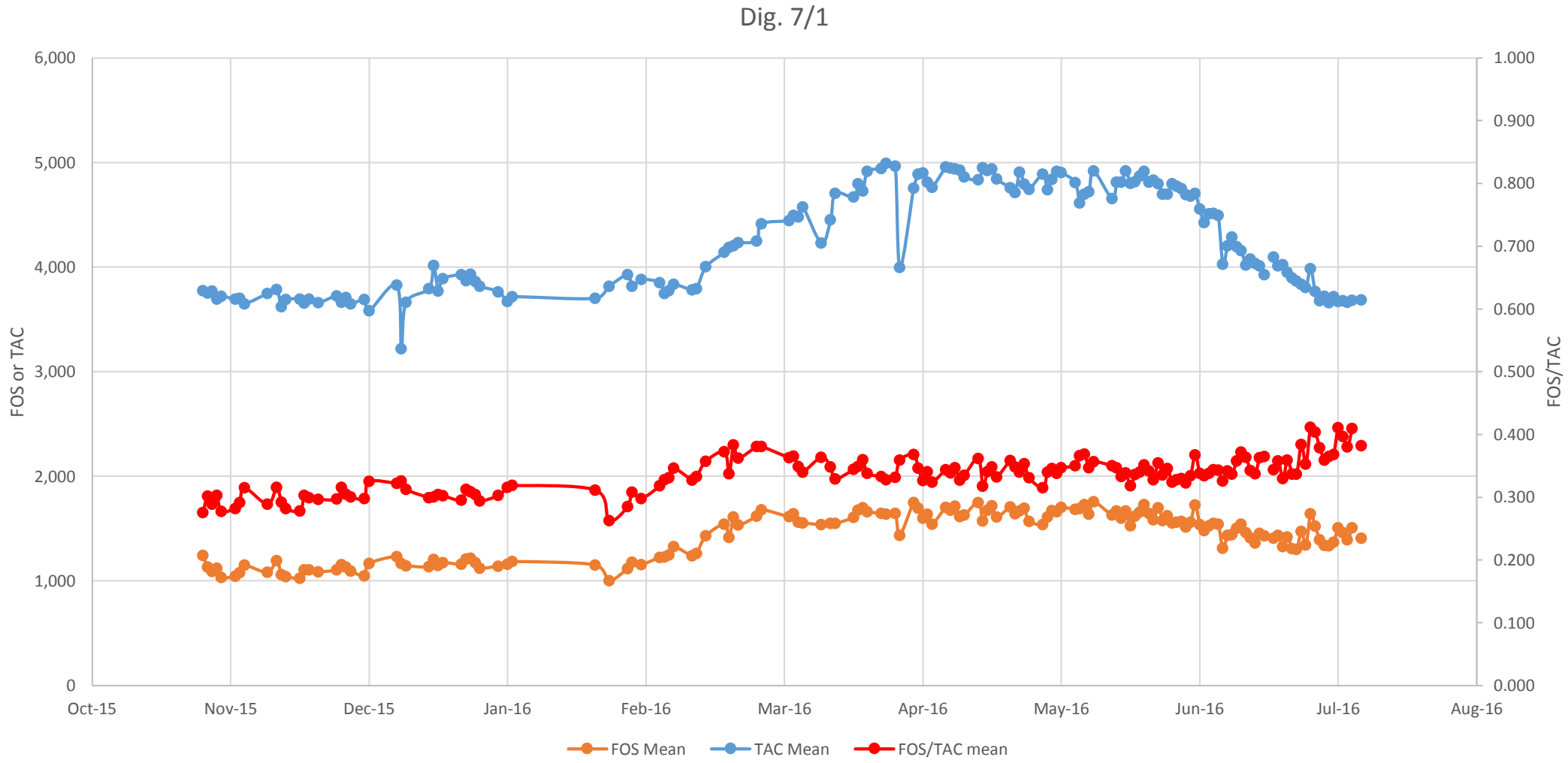
Lab Results FOS



Lab Results TAC

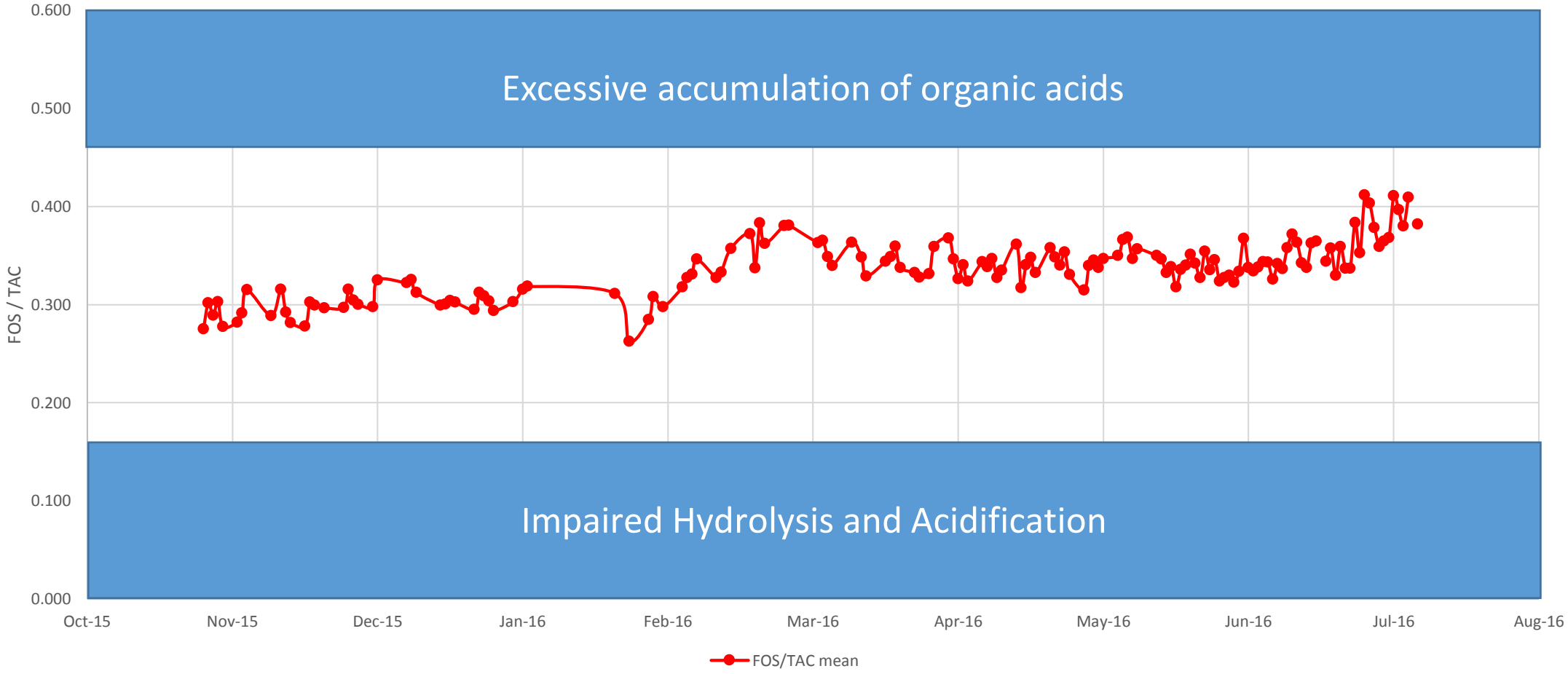


Digester 7/1 Data

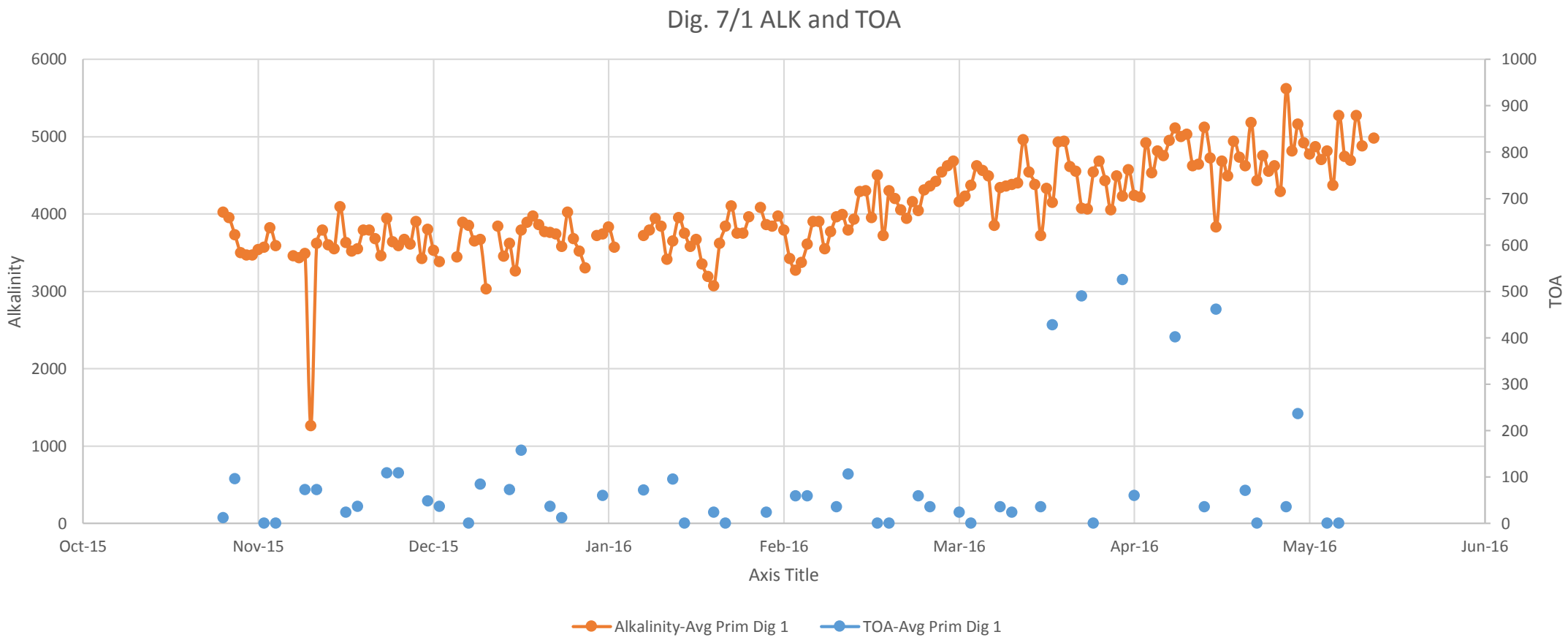


FOS/TAC in Process Control

Dig. 7/1



Old Data


Dig. 7/1 ALK and TOA
Alkalinity
TOA
Axis Title
6000
5000
4000
3000
2000
1000
0
1000
900
800
700
600
500
400
300
200
100
0
Oct-15
Nov-15
Dec-15
Jan-16
Feb-16
Mar-16
Apr-16
May-16
Jun-16
Alkalinity-Avg Prim Dig 1
TOA-Avg Prim Dig 1

Thank you, for your attention.

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Thank you!

UOSA-Laboratory staff
and
UOSA-TP Process Analysis staff

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Reference Literature:

FOS/TAC – Deduction, Methods, Application and Significance Voß, E., Weichgrebe D. Rosenwinkel, K.-H.

Determination of FOS/TAC Value in Biogas Reactors Hach DOC316.52.93087

A comparison of two simple titration procedures to determine volatile fatty acids in influents to waste-water and sludge treatment processes Buchauer, K